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ABSTRACT:

CHG DATE=19990617 STATUS=O> The interfacial surface tension between two immiscible liquids is measured by taking a sample of one liquid into a vertical capillary tube 5 whose lower end is then immersed in a body of the second liquid 12. The top of tube 5 is connected to a large closed volume of air 1, a pressure monitor 10 able to measure pressure to an accuracy of 0.01 m Bar and to a syringe 2 whose plunger 4 is movable by a screw to vary the pressure in system 1, 5, 10. The pressure is thus varied while the meniscus at the lower end of tube 5 is observed. The pressure difference recorded between two

states, when the meniscus is flat and when it is hemispherical, divided by the capillary tube radius gives a measure of the surface tension. Tube 5 is glass with a circular section bore and a square section exterior, the outer surfaces and the end being optically flat. The bore is 0.5 mm diameter. The meniscus is observed through windows in vessel 13 using a lens system or-TV camera. The temperature in vessel 13 is controlled by means 14. In Fig. 2 the vessel is closed and the pressure monitor records the difference between pressures inside the capillary and inside the vessel above the liquid level. By measuring the surface tension between an oil sample and aqueous solutions containing various amounts of surfactants, a graph is plotted whose trough indicates the HLB requirement of the oil, enabling a suitable surfactant to be selected for making an oil-water emulsion. <IMAGE> .

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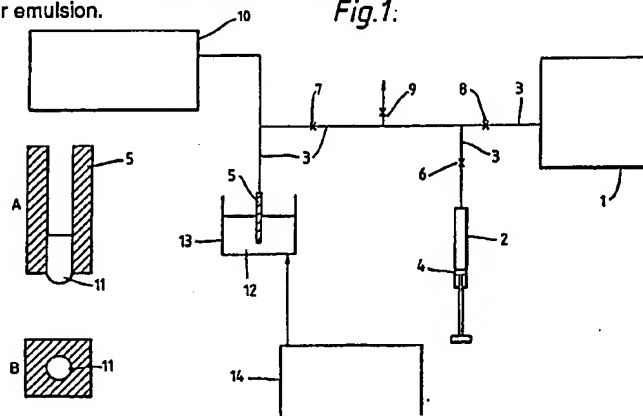
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(54) Measurement of Interfacial surface tension

(57) The interfacial surface tension between two immiscible liquids is measured by taking a sample of one liquid into a vertical capillary tube 5 whose lower end is then immersed in a body of the second liquid 12. The top of tube 5 is connected to a large closed volume of air 1, a pressure monitor 10 able to measure pressure to an accuracy of 0.01 m Bar and to a syringe 2 whose plunger 4 is movable by a screw to vary the pressure in system 1, 5, 10. The pressure is thus varied while the meniscus at the lower end of tube 5 is observed. The pressure difference recorded between two states, when the meniscus is flat and when it is hemispherical, divided by the capillary tube radius gives a measure of the surface tension. Tube 5 is glass with a circular section bore and a square section exterior, the outer surfaces and the end being optically flat. The bore is 0.5 mm diameter. The meniscus is observed through windows in vessel 13 using a lens system or TV camera. The temperature in vessel 13 is controlled by means 14. In Fig. 2 the vessel is closed and the pressure monitor records the difference between pressures inside the capillary and inside the vessel above the liquid level.

By measuring the surface tension between an oil sample and aqueous solutions containing various amounts of surfactants, a graph is plotted whose trough indicates the HLB requirement of the oil, enabling a suitable surfactant to be selected for making an oil-water emulsion.

Fig.1:



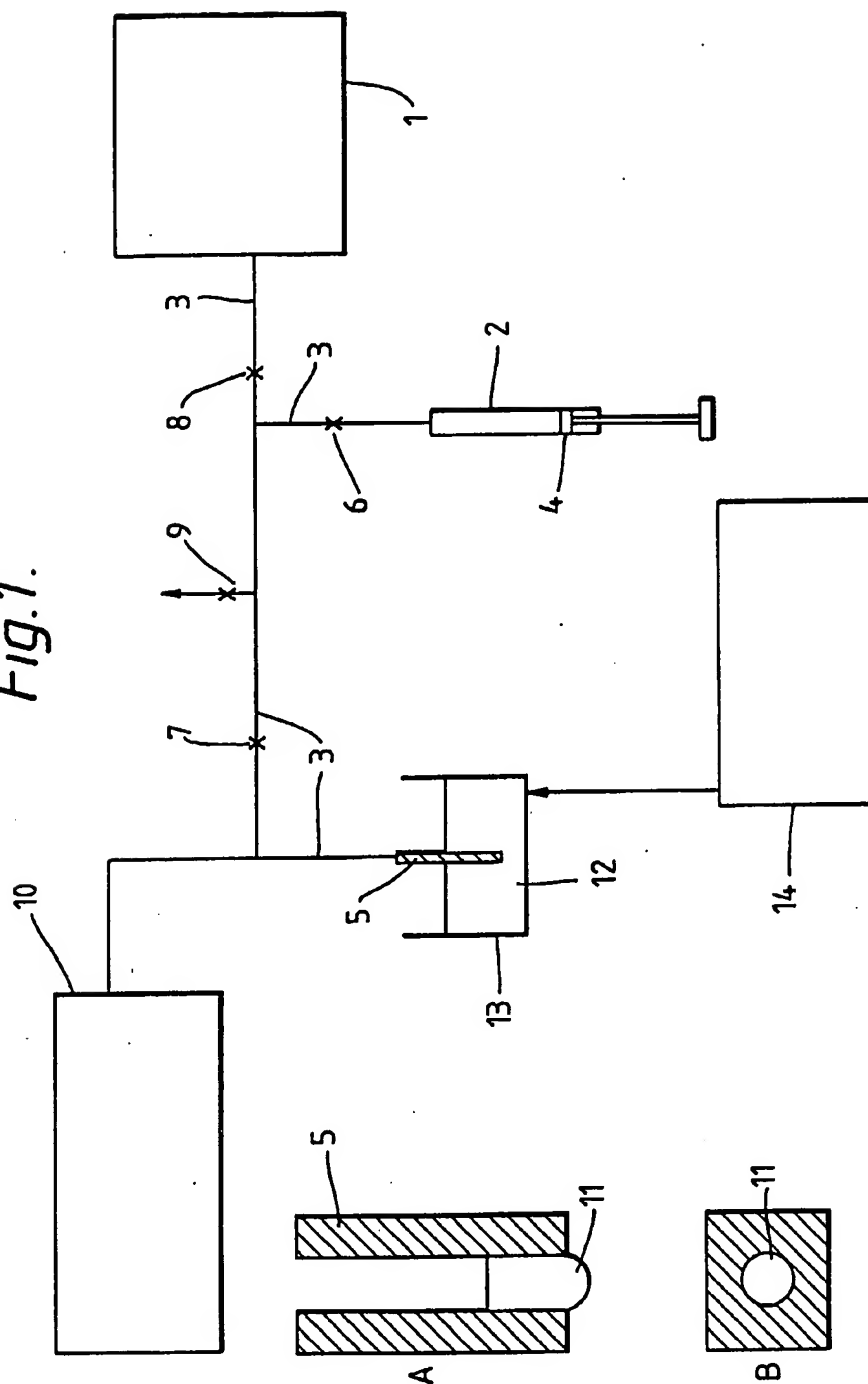
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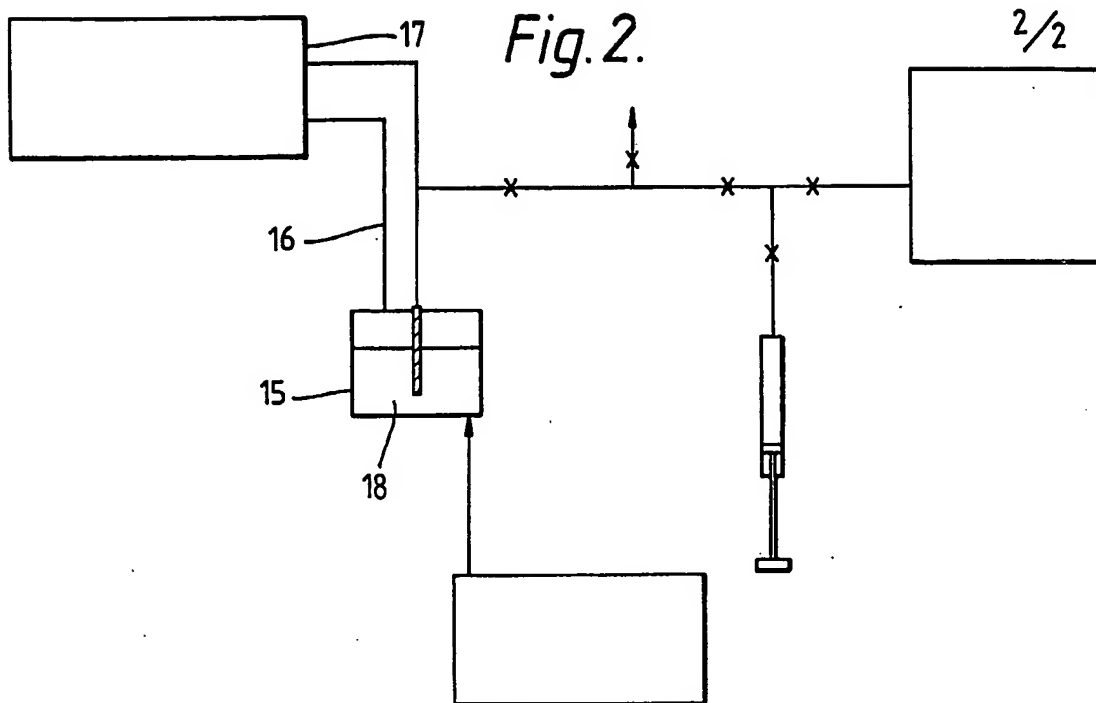
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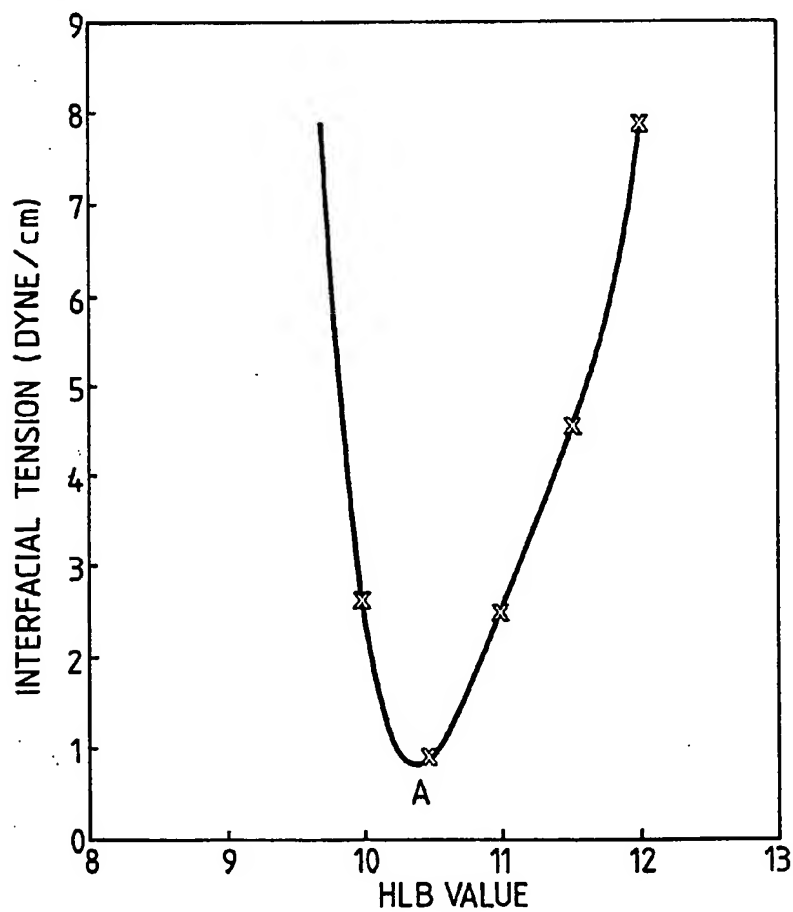
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Fig.1.





*Fig. 3.*



MEASUREMENT OF INTERFACIAL SURFACE TENSION

The present invention relates to an apparatus and method for the precise measurement of the interfacial surface tension between two immiscible liquids at values in the region of 0.001 to 80 mN/m with an accuracy of at least 0.001 mN/m, and having also the advantage of ease of operation, the avoidance of need for calibration or application of corrections to the observed parameters. In particular, the apparatus and method are especially advantageous in requiring samples of one of the liquids, for example an oil, and any additives such as a surfactant used therewith, in very small quantities.

In formulating emulsions with oil and water to make stable water in oil or oil in water emulsions there are practical problems. For example, it is common practice to identify surfactants or mixtures of surfactants that may be used for making emulsions in an empirical way. A large number of mixtures of surfactants of different generic type are used to make emulsions of oil and water and the stability of the resulting formulation assessed visually by allowing the formulations to stand and observing the rate at which the emulsions break. There is a practical difficulty in ensuring that all the formulations are treated in exactly the same manner with respect to the shaking and emulsification and there is often a practical problem in differentiating the stability performance of emulsions with similar short term appearance which may not be reflected in the longer time scale. The emulsifier can be conveniently characterised by reference to its hydrophilic - lipophilic balance (HLB) value. This is an arbitrary scale which defines the balance of the hydrophilic and lipophilic groups present in the emulsifier and therefore the propensity of the emulsifier to dissolve in oils and water and in consequence its activity at an oil - water interface. In a similar manner the lipophilic character of an oil may be described in terms of its HLB oil requirement. When the hydrophilic - lipophilic balance oil requirement is known it is possible to choose only those surfactants or surfactant mixtures that produce HLB values that approximate to the HLB oil requirement.

We have now found a new, practical and more precise method by which the labour and effort required to identify candidate surfactants or mixtures of candidates can be greatly diminished.

According to the present invention an apparatus for measuring interfacial surface tension between two immiscible liquids comprises an open-ended transparent capillary tube having a circular bore and adapted to be held in a vertical position, said capillary tube being adapted to contain a first liquid and mounted so that the lower end of the capillary tube is adapted to be immersed in a second liquid; a normally closed ballast vessel inter-connected by air-tight conduits to a pressure measuring means capable of measuring pressure to an accuracy of at least 0.01mBar, to the upper end of the capillary tube and to means adapted to vary in a regulated amount, the ballast volume of the apparatus thereby enclosed in air-tight manner; valve means adapted to isolate the capillary and/or the pressure measuring means from the ballast vessel, and viewing means adapted to observe the first liquid in the capillary tube at the point where it makes contact with the second liquid.

The apparatus according to the invention may additionally comprise valve means adapted to vent the ballast vessel to the atmosphere.

The means adapted to vary the ballast volume of the apparatus is preferably provided by a number, conveniently three, syringes of different sizes, for example 50ml, 10ml and 1ml, fitted with gas-tight plungers, conveniently mounted such that the extent of the travel of the plungers can be regulated by means of a knurled knob attached to a threaded rod.

The greater part of the total volume of the apparatus is conveniently provided by a ballast vessel such as a 5 litre reaction flask which acts as a ballast volume. The size of the ballast vessel is chosen so that convenient changes in the total ballast volume leads to changes in the pressure in the apparatus in a range suitable for changing the shape of the interface between the immiscible liquids as hereinafter described.

The capillary is preferably made of glass preferably with a precision circular bore of 0.50mm, but other dimensions may be used provided that the precise diameter of the tube is known and provided that the choice thereof permits convenient measurement in the appropriate range of surface tension values. The exterior of the lower part of the capillary tube is preferably of square

cross-section, and the surfaces thereof made optically flat in order to eliminate a lens effect when viewing the capillary and to permit the direct measurement of the bore with a travelling microscope without the need for correction. The base of the capillary tube is also preferably ground optically flat.

The pressure measuring means may suitably be a capacitance manometer pressure sensor in which diaphragms form part of a capacitor in a bridge circuit. These diaphragms become distorted when subjected to pressure differences and the consequent change in capacitance can be detected by electronic circuitry with great accuracy. The pressure measuring means is preferably apparatus which measures pressure absolutely, or differences in pressure absolutely, and capable of measuring pressures of about atmospheric to hundredths of a millibar or less. Equipment provided with a digital readout in millibar or other preferred units is convenient.

The second liquid, normally an aqueous phase in which the lower end of the capillary tube carrying a thread of the first liquid, normally an oil phase, is immersed, is contained in a second vessel the temperature of which may be thermostatically controlled for example by a temperature measuring device in the second liquid which controls heaters in the walls of the said vessel or in any other conventional manner. The said vessel is preferably constructed with four vertical walls each fitted with a window to permit the illumination of the capillary tube by means of a suitable light source and thereby to facilitate the observation of the first liquid in the capillary tube where it makes contact with the second liquid. Such observation may conveniently be made by eye, with the aid of a lens, with the aid of a low power microscope, by projecting an image of the capillary onto a screen or more conveniently by using a television camera and a suitable lens system and observing the image on a television monitor.

The conduits inter-connecting the ballast vessel, the pressure measuring means, the capillary tube and the means for varying the ballast volume may conveniently comprise metal, glass or plastic tubing.

The invention also comprises a method for the measurements of interfacial surface tension between two immiscible liquids comprising the steps of;

- i taking up in small volume of the first liquid in a capillary tube of known radius;
- ii mounting the capillary tube vertically in a volume of the second liquid so that the lower part of the capillary tube is immersed in the second liquid;
- iii connecting the open upper end of the capillary tube to a comparatively large ballast volume of air;
- iv altering the ballast volume in a precise manner to, adjust the pressure within the capillary tube and ballast vessel so that the interface between the first liquid and the second liquid is flat, (i.e. of infinite radius) and noting the pressure P<sub>1</sub>;
- v further altering the ballast volume in like manner to adjust the pressure within the capillary tube and the ballast vessel so that the interface between the first liquid and the second liquid is hemispherical and noting the pressure P<sub>2</sub>; and
- vi deducing the interfacial surface tension by applying the formula

$$\text{surface tension} = \frac{R(P_2 - P_1)}{2}$$

2

where R is the radius of the capillary, P<sub>2</sub>, P<sub>1</sub> and R to be expressed in consistent units.

The arrangement of valve means conveniently permits the capillary tube to be isolated from the ballast vessel and, when an appropriate valve means is used, for the ballast vessel to be opened to the atmosphere. Such arrangements permit the increase and decrease of pressure within the apparatus beyond that which can conveniently be achieved by operation of the preferred means adapted to vary the ballast volume of the apparatus, namely the syringe plungers over their range, by isolating the capillary tube and the ballast vessel from the syringe, opening the syringe to atmosphere, moving the syringe plunger to the opposite end of its travel, closing the valve means to atmosphere, and

re-opening the valve means to the capillary tube and the ballast vessel and repeating this process as often as required.

The invention also provides a method to aid the selection of an emulsifier formulation for the optimum composition of emulsifier required to prepare a stable emulsion of oil phase in aqueous phase or an aqueous phase in oil phase, or an emulsion of two essentially immiscible liquids one within the other, which comprises the measurement of the interfacial surface tension between the oil phase and an aqueous phase (or between the two immiscible fluid phases) as a function of the amounts and chemical identities of the surfactant species present, to determine the conditions which provide the minimum interfacial surface tension to enable the matching or close matching of the HLB oil requirement to the HLB value of the surfactant or surfactant mixture.

Examples of the present invention will now be described with reference to the accompanying drawings in which

Figure 1. is a sketch illustrating the features of an apparatus according to the invention.

Figure 2. is a sketch illustrating additional features of the apparatus shown in Figure 1. modified in respect of the manner of the pressure measurement.

Figure 3. is a graph constructed from the measurements obtained in Example 2. as an aid for their interpretation.

In one embodiment of the apparatus according to the invention the component parts are assembled as illustrated in Figure 1. to which reference is made in the following description.

The greater part of the total volume of the apparatus shown on Figure 1. is provided by ballast vessel (1). The minor part of the total volume of the apparatus is made up of a syringe (2) and the connecting tubing and pipework (3). The syringe (2) is fitted with a gas-tight plunger (4). Syringe (2) is connected to capillary tube (5) via two valves (6, 7) normally open to the ballast vessel (1) via a valve (8) normally open and to a valve (9) which opens to the atmosphere, but is normally closed. Capillary (5) is made of glass and has a precision circular bore of 0.50mm. The exterior of the lower part of capillary tube (5) is of square cross-section as shown in inset B, the surfaces thereof being optically flat. The base of the capillary (5) is ground optically flat. The open upper end of capillary (5) is

connected to an MKS Instruments Baratron Type 170M-35 capacitance manometer. Such equipment may be purchased from Chell Instruments Ltd., North Walsham, Norfolk. The equipment is provided with a digital readout of the pressure in millibar or other preferred units.

The capillary (5) contains a thread of oil phase (11). The capillary (5) is mounted such that its lower end is immersed in an aqueous phase (12) contained in a vessel (13) the temperature of which is thermostatically maintained by a controlling apparatus (14). The controlling apparatus (14) controls heaters within the walls of vessel (13) with reference to a thermocouple or other temperature sensing device appropriately positioned in the liquid (12). The vessel (13) is preferably made with four vertical walls each with a window to permit the illumination of the capillary tube (5) with a light source to observe the thread of the oil phase (11) in the capillary (5) where it makes contact with the aqueous phase (12). Such observations were made by using a television camera and a suitable magnifying lens system and observing the image on a television monitor.

The preferred method of operation requires that the pressure inside the apparatus be increased so that the meniscus between the fluid (11) in the capillary (5) and the liquid phase (12) in the vessel (13) is flat, corresponding to pressure  $P_1$  and then to a pressure such that the meniscus is hemispherical corresponding to pressure  $P_2$ . Because the pressure inside the expressed bubble is a maximum when its radius is equal to the radius of the capillary, for at other conditions the radius is greater than that of the capillary, it is most convenient to gradually reduce the ballast volume by altering the syringe (2) whilst observing the digital read-out of an electronic pressure measuring device (10) and noting when it passes through a maximum pressure. It is necessary to have the pressure reading corresponding to the situation when the meniscus is flat and this can be found by altering the ballast volume with the syringe (2) until a flat interface can be seen visually, or preferably with the aid of the television camera and lens system.

The arrangement of valves is such as to permit the pressure within the apparatus to be increased or decreased beyond that which can conveniently be achieved by operating the syringe plunger (4) over its range of travel. This may be done by closing valves (7) and (8), opening valve (9), moving the plunger to the opposite end of its travel,

closing valve (9) and re-opening valves (7) and (8). The purpose of valve (6) is to provide a means for checking for leaks and it may also be used to change the syringe (2) without substantially changing the pressure within the apparatus.

In a second embodiment of the invention the apparatus is modified as illustrated in Figure 2 wherein the pressure measuring device (17) measures a differential pressure by being connected to the capillary as before, and also to the space above an enclosed vessel (15) containing the fluid (18) by a pipe (16). By this means a more sensitive measurement can be made as transient local adventitious fluctuations in atmospheric pressure are eliminated. Such fluctuations may arise from convective or other draughts in the vicinity of the vessel containing the aqueous phase and carrying the capillary, especially when it is desired to make measurements at temperatures other than room temperature when the vessel is deliberately heated or cooled. The measurement of pressure differences of the order of less than one hundredth of a millibar may be practically difficult in the absence of such a precaution.

The range of values most appropriate for the apparatus of the invention is, at the higher end, up to at least 72 mN/m (or dynes/cm) which is the value of air against water, down to fractions of a mN/m (or dyne/cm). It is practical to measure down to 0.5 mN/m with a pressure measuring device that registers 0.01mBar examples with about 0.3 mN/m precision, but with a pressure measuring device capable of measuring to one or two orders of magnitude lower than this then proportionately lower interfacial surface tensions can be measured.

The apparatus of the invention is especially useful when assessing the effect of surface active agents and particularly emulsifying agents in lowering the interfacial surface tension between an oil and an aqueous phase where the oil and aqueous phase are essentially immiscible. It may, for example, be used to determine the hydrophilic-lipophilic balance (HLB) of an unknown surfactant by using an oil of known HLB oil requirement in conjunction with a surfactant of known HLB value, or vice versa be used to determine the HLB oil requirement of an unknown oil by using surfactants of known HLB values. The effects of temperature, concentration of surfactant, added electrolyte to the aqueous phase, and the addition of other chemical

species that alter the HLB of the system, may also be determined. The surfactant(s) may be conveniently added to either the oil phase or the aqueous phase or to both depending upon the solubilities of the surfactants in the two phases. The effective HLB oil requirement of ideal solutes in an oil of known HLB oil requirement can also be deduced by following the same basic procedure, and this is particularly advantageous where the solute is a viscous oil whose interfacial surface tension against water or air is otherwise practically difficult or inconvenient by other methods.

The apparatus may also be used to measure the surface tension of liquids against air (or other gaseous phase when the enclosed configuration is adopted).

Because of the simple and rapid nature of the measurement, it is also possible to study changes with time in the interfacial surface tension between two immiscible fluids in, for example, circumstances where the equilibration of the distribution of a surfactant between the two phases is comparatively slow (of the order of minutes).

It also has the advantage of using only small volumes of oil phase which means that the system comes to a more rapid concentration equilibrium and may be particularly useful where the amount of test material available is severely limited.

It is also of practical value where an unknown oil, which is viscous and for this reason it is practically difficult to measure the interfacial surface tension against air or an aqueous solution, when it can be incorporated as an ideal solute in an oil of known HLB oil requirement, measuring the manner in which the HLB oil requirement of the oil is altered by incorporating the unknown as a solute and extrapolating the observations to that expected for the unknown oil alone.

The invention is illustrated but not limited by the following examples of the application of the apparatus according to the invention.

Example 1.

The apparatus was used in a configuration in which the total ballast volume was 5.745 litres, the capillary was 0.50mm in diameter, and there were available to adjust the volume a 50ml, a 10ml and 1ml syringe. A small volume of n-octanol was introduced to the capillary by touching the surface of some n-octanol liquid and allowing capillary forces to draw a thread of liquid into the capillary. The capillary was then connected to the ballast volume and mounted so that the tip of the capillary was below the level of distilled water contained in a stainless steel vessel fitted with windows. By adjusting the syringes and operating the valves the pressure inside the capillary was made such that the interface between the n-octanol and the water was flat. The difference in volume required to increase this pressure to the point where the interface became hemispherical was noted. This difference corresponded to 1.8mls and by calculation this leads to a value for the interfacial surface tension of  $8.0 \pm 0.3$  dynes/cm (mN/m) which is in accord with the accepted value.

Example 2.

In this example two surfactants were used of known HLB value to deduce the HLB oil requirement of an unknown oil. SYNPERONIC PE/L42 is a propylene oxide/ethylene oxide block copolymer in which the ethylene oxide content is about 20%. It has an HLB value of 8.0. SYNPERONIC PE/L64 is a propylene oxide/ethylene oxide block copolymer in which the ethylene oxide content is about 40%. It has an HLB value of 15.0. Both of these surfactants are soluble in water at moderate concentrations. SYNPERONIC PE is a trade name of ICI plc and both surfactants may be obtained from ICI plc.

A light white mineral oil of density 0.830-0.850g/cc and a viscosity at 40°C of 10-14 centi-stokes was used.

A series of aqueous solutions was made containing different concentrations of the two surfactants so that the HLB of the resultant

aqueous solutions covered the range between 8.0 and 15.0 as calculated from the weight percent proportion of each surfactant present. These solutions were diluted to 1wt% of total surfactant and used as the aqueous phase in a series of determinations of the interfacial surface tension against the mineral oil held in the capillary. The interfacial surface tension values were then plotted as a function of the HLB value as shown in Figure 3., and a curve fitted to the observed data. It was deduced that the position of the minimum (A) interfacial surface tension corresponding to the HLB oil requirement occurred at  $10.4 \pm 0.1$ .

The principle of the method may be used to deduce the HLB value of an unknown surfactant where the HLB value of another differently valued surfactant and an oil of known HLB oil requirement are available.

## Claims

- 1 Apparatus for measuring interfacial surface tension between two immiscible liquids comprises an open-ended transparent capillary tube having a circular bore and adapted to be held in a vertical position, said capillary tube being adapted to contain a first liquid and mounted so that the lower end of the capillary tube is adapted to be immersed in a second liquid; a normally closed ballast vessel inter-connected by air tight conduits to a pressure measuring means capable of measuring pressure to an accuracy of at least 0.01mBar, to the upper end of the capillary tube and to means adapted to vary in a regulated amount, the ballast volume of the apparatus thereby enclosed in air tight manner; valve means adapted to isolate the capillary and/or the pressure measuring means from the ballast vessel, and viewing means adapted to observe the first liquid in the capillary tube at the point where it makes contact with the second liquid.
- 2 Apparatus as claimed in claim 1 which additionally comprise valve means adapted to vent the ballast vessel to the atmosphere.
- 3 Apparatus as claimed in either claim 1 or claim 2 in which the means adapted to vary the ballast volume of the apparatus is preferably provided by a number of syringes of different sizes fitted with gas-tight plungers mounted such that the extent of the travel of the plungers can be regulated by means of a knurled knob attached to a threaded rod.
- 4 A method for the measurements of interfacial surface tension between two immiscible liquids comprising the steps of;
  - i taking up in small volume of the first liquid in a capillary tube of known radius,
  - ii mounting the capillary tube vertically in a volume of the second liquid so that the lower part of the capillary tube is immersed in the second liquid,

- iii connecting the open upper end of the capillary tube to a comparatively large ballast volume of air,
- iv altering the ballast volume in a precise manner to, adjust the pressure within the capillary tube and ballast vessel so that the interface between the first liquid and the second liquid is flat, (i.e. of infinite radius) and noting the pressure P1,
- v further altering the ballast volume in like manner to adjust the pressure within the capillary tube and the ballast vessel so that the interface between the first liquid and the second liquid is hemispherical and noting the pressure P2 and
- vi deducing the interfacial surface tension by applying the formula

$$\text{surface tension} = \frac{R(P_2 - P_1)}{2}$$

2

where R is the radius of the capillary, P2, P1 and R to be expressed in consistent units.

**Amendments to the claims have been filed as follows**

- 1 Apparatus for measuring interfacial surface tension between two immiscible liquids comprises an open-ended transparent capillary tube having a circular bore and adapted to be held in a vertical position, said capillary tube being adapted to contain a first liquid and mounted so that the lower end of the capillary tube is adapted to be immersed in a second liquid; a normally closed ballast vessel inter-connected by air tight conduits to a pressure measuring means capable of measuring pressure to an accuracy of at least 0.01mBar, to the upper end of the capillary tube and to means adapted to vary in a regulated amount, the ballast volume of the apparatus thereby enclosed in air tight manner; valve means adapted to isolate the capillary and/or the pressure measuring means from the ballast vessel, and viewing means adapted to observe the first liquid in the capillary tube at the point where it makes contact with the second liquid.
- 2 Apparatus as claimed in claim 1 which additionally comprise valve means adapted to vent the ballast vessel to the atmosphere.
- 3 Apparatus as claimed in either claim 1 or claim 2 in which the means adapted to vary the ballast volume of the apparatus is preferably provided by a number of syringes of different sizes fitted with gas-tight plungers mounted such that the extent of the travel of the plungers can be regulated by means of a knurled knob attached to a threaded rod.
- 4 A method for the measurements of interfacial surface tension between two immiscible liquids comprising the steps of;
  - i taking up in small volume of the first liquid in a capillary tube of known radius,
  - ii mounting the capillary tube vertically in a volume of the second liquid so that the lower part of the capillary tube is immersed in the second liquid,

- iii connecting the open upper end of the capillary tube to a comparatively large ballast volume of air,
- iv altering the ballast volume in a precise manner to, adjust the pressure within the capillary tube and ballast vessel so that the interface between the first liquid and the second liquid is flat, (i.e. of infinite radius) and noting the pressure P1,
- v further altering the ballast volume in like manner to adjust the pressure within the capillary tube and the ballast vessel so that the interface between the first liquid and the second liquid is hemispherical and noting the pressure P2 and
- vi deducing the interfacial surface tension by applying the formula

$$\text{surface tension} = \frac{R(P_2 - P_1)}{2}$$

2

where R is the radius of the capillary, P2, P1 and R to be expressed in consistent units.

- 5 A method of selecting a surfactant formulation for the optimum composition of emulsifier required to prepare a stable emulsion of two essentially immiscible liquids one within the other, which comprises the measurement of the interfacial surface tension between the two immiscible liquid phases, using the apparatus claimed in any one of claims 1 to 3 or apparatus substantially as herinbefore described, or by the method claimed in claim 4 or the method substantially as herinbefore described, in the presence of the surfactant formulation, as a function of the amounts and chemical identities of the components of the surfactant formulation, to determine the conditions which provide the minimum interfacial surface tension to enable the matching or close matching of the HLB oil requirement to the HLB value of the surfactant formulation.
- 6 A method as claimed in claim 5 wherein the immiscible liquids are an aqueous phase and an oil phase.

**Patents Act 1977**  
**Examiner's report to the Comptroller under**  
**Section 17 (The Search Report)**

15

Application number

GB 9306132.3

**Relevant Technical fields**

(i) UK CI (Edition L ) G1S (SKA)

(ii) Int CI (Edition 5 ) G01N (13/00, 13/02)

**Databases (see over)**

(i) UK Patent Office

(ii)

**Search Examiner**

R T HAINES

**Date of Search**

24 MAY 1993

Documents considered relevant following a search in respect of claims 1-4

Category (see over)	Identity of document and relevant passages	Relevant to claim(s)
	NONE	

SF2(p)

me - doc99\fil000595

Category	Identity of document and relevant passages	Relevant to claim(s)

#### Categories of documents

X: Document indicating lack of novelty or of inventive step.

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